Environmental and In Situ SEM/TEM

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X-ray microanalysis in the environmental or variable pressure scanning electron microscope

J. Rattenberger[1], H. Schroettner[1,2], J. Wagner[1], F. Hofer[1,2]

[1]Centre for Electron Microscopy, Graz, Austria
[2]Graz University of Technology, Institute of Electron Microscopy and Nanoanalysis, Graz, Austria

johannes.rattenberger@felmi-zfe.at
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Environmental or variable pressure scanning electron microscopy enables the opportunity to investigate uncoated insulators, organic, biological or even wet samples in their original state. The drawback of this technique is the scattering of primary beam electrons inside the gaseous environment of the specimen chamber, which degrades the signal to noise ratio and complicates X-ray spectrometry.

The trajectories of the scattered primary beam electrons are deflected and characteristic X-rays which originate from the surrounding but not interesting area are detected. In order to demonstrate this effect a segregation area in a steel sample was investigated under low vacuum and high vacuum conditions (see figure 1). The beam was focused on a segregation area in steel and the spectra were compared. The iron signal is strongly reduced in comparison with the high vacuum spectrum. Therefore, additional correction procedures are necessary for an accurate quantification of such spectra.

There are two basic different correction procedures, the beam stop procedure and the pressure variation procedure. The beam stop procedure is unfunctional because a micromanipulator needle made of a well-known element is needed [1].

The pressure variation method needs no additional equipment and is therefore much more practically orientated [2,3]. To use this procedure the X-ray intensities must be measured for two different chamber pressure conditions. By calculating the fraction of unscattered electrons for both pressures the unaffected intensity at 0 Pascal chamber pressure can be calculated.

The major challenge using this correction method is calculating the fraction of unscattered electrons. Therefore, the total scattering cross section of the imaging gas as well as detailed knowledge of the interaction distance between primary beam electrons and the imaging gas is necessary.

Usually the beam transfer characteristic of the microscope is neglected, which leads to an inaccuracy in measurement because the working distance (distance between pole piece and specimen) is used as interaction distance and not the stagnation gas thickness (□) which considers the static gas flow from the specimen chamber upwards the electron column and the position of the pressure limiting aperture [4]. This distance is depending on the microscope, pressure difference and gas type (see figure 2 and figure 3). Approximations and simplifications in calculating the total scattering cross section lead to additional measurement inaccuracies.

An optimised method and more precise results are presented by considering the beam transfer characteristic and using experimentally measured total scattering cross sections.

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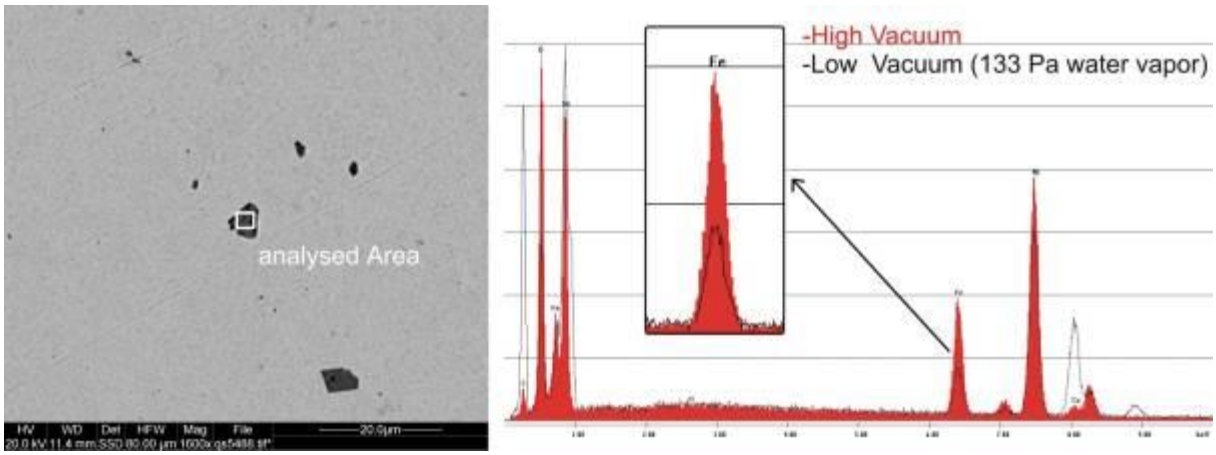

Figure 1. Analyzed area (left); Comparison of EDX spectra (low vacuum and high vacuum) (right)

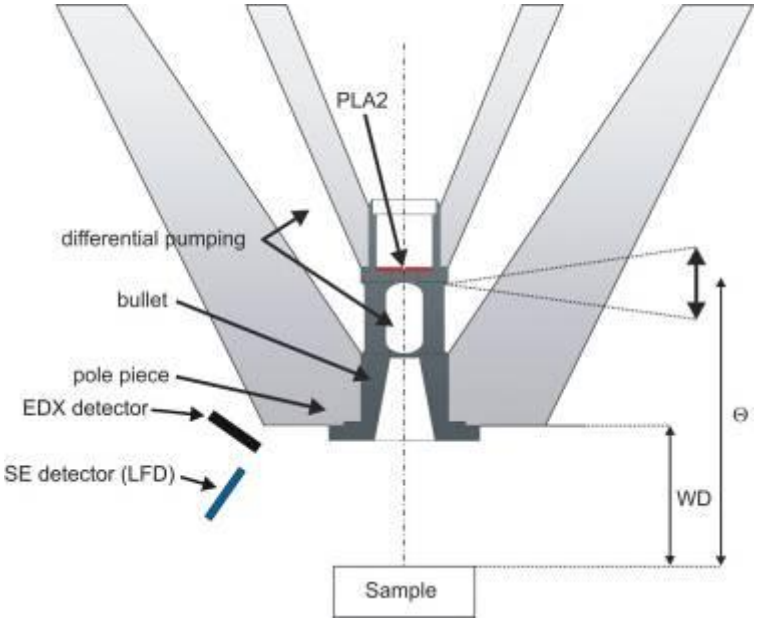

Figure 2. Schematic drawing of the FEI ESEM Quanta 600

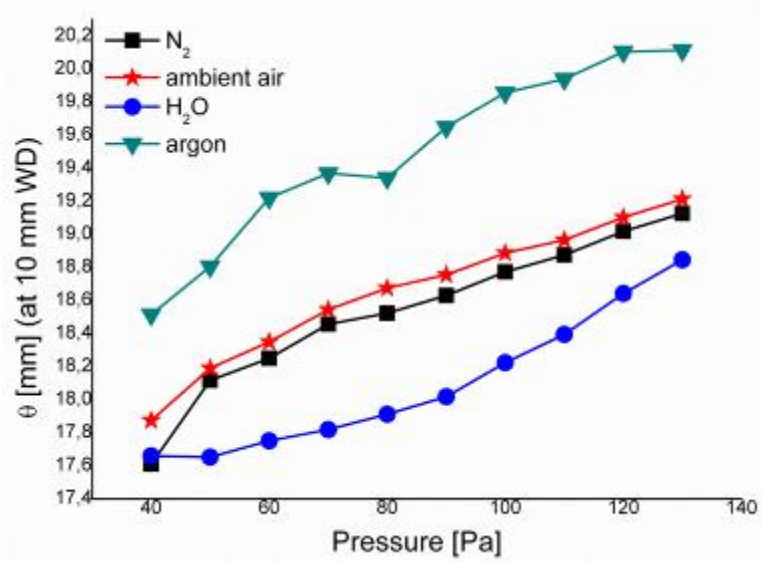

Figure 3. Stagnation gas thickness (□) [mm] as a function of the chamber pressure [Pa]